IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Appl. No.	:	10/551,854	Confirmation No. 6166
Applicant (s)	:	Zenon Lysenko, et al.	
Filed	:	September 30, 2005	
TC/A.U.	:	1621	
Examiner	:	Cutliff, Yate Kai Rene	
Title	:	ALDEHYDE AND ALCOHOL COMPOSITIONS DERIVED FROM SEED OILS	
Docket No.	:	63104A	
Customer No.	:	00109	

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

DECLARATION II UNDER 37 C.F.R. §1.132

David A. Babb declares and states:

THAT, he and Zenon Lysenko, Donald L. Morrison, Donald L. Bunning, Christopher W. Derstine, James H. Gilchrist, H. Ray Jouett, Jeffrey S. Kanel, Kurt D. Olson, Wei-Jun Peng, Joe D. Phillips, Brian R. Roesch, Aaron W. Sanders, Alan K. Schrock, and Pulikkottil J. Thomas are the inventors of the subject technology of the above-captioned patent application, and that he is familiar with the Office Action dated January 31, 2008, in the above-identified application;

THAT, in 1982 he received a Bachelors of Science degree in Chemistry from Texas Tech University, and in 1985 he received a Ph.D. degree in Chemistry from Texas Tech University;

THAT, from 1985 until the current date he has worked at The Dow Chemical Company as Research Chemist (1985-1993), as Research Leader (1993-2004), and in his current position as Research Scientist (2004 – present), where he now leads a project to manufacture and evaluate polyurethanes prepared from polyols derived from seed oils;

THAT, the following experiments were designed by him and conducted under his supervision for the purpose of preparing a monomer alcohol composition having a diol/triol weight ratio greater than 5/1 illustrative of the claimed invention, and for the

purpose of using the monomer alcohol to prepare a polyol, which itself was used to prepare a polyurethane suitable for flexible foam applications.

Materials for Use in Preparing Polyol

An alcohol monomer composition having a diol/triol weight ratio of 5.56/1 and comprising a mixture of hydroxymethylated fatty acid methyl esters was produced by hydroformylation and reduction of a mixture of unsaturated sunflower oil methyl esters in the manner described in the above-captioned application, with details given hereinafter.

INT-1 polyol, obtained from The Dow Chemical Company, is a glycerin initiated polyethylene oxide (EO) polyol with a number average molecular weight of 620 and a hydroxyl number of 267.

Tin (II) ethylhexanoate, as product DABCO T-9, was obtained from Air Products & Chemicals, Inc.

Example 1 - Preparation of a Polyol Using a Monomer Alcohol of the Invention

This example illustrates preparation of a polyol derived from a monomer alcohol having a diol/triol weight ratio greater than 5/1 within the scope of the claims, specifically, a diol/triol weight ratio of 5.56/1.

With reference to Applicants' specification, sunflower oil comprising a mixture of unsaturated fatty acid methyl esters was hydroxymethylated to form a sunflower-based monomer alcohol according to the description and examples of the invention. The hydroformylation conditions were similar to those of Example 2 of the present application. The resulting monomer aldehyde comprising a mixture of formyl-substituted sunflower oil methyl esters was hydrogenated under hydrogen in the manner described in the invention, specifically, in the presence of a supported nickel catalyst (Sud-Chemie C-46-8-03) at 120°C and 400 psig (2557 kPa) hydrogen gas to obtain the monomer alcohol having a diol/triol weight ratio of 5.56/1. The composition of the sunflower-based monomer alcohol is shown in Table 1.

Table 1 - Monomer Alcohol from Sunflower Oil

Component	Example 1
	Weight %
Saturates	7.90
Monols	83.98
Diols	5.00
Triols	0.90
Lactols/Cyclic ethers	1.31
Lactones	0.35
Others (including dimers)	0.52
Total	100.00
Diol/Triol	**5.56/1**

The above-identified monomer alcohol (37.875 kg) was charged to a 30 gallon (114 l) stainless steel jacketed reactor vessel together with INT-1 initiator (17.050 kg). The reactor vessel was equipped with a nitrogen sparger, an agitator, a turbine for gas dispersion, a vacuum system, and hot oil as a heating medium. The mixture was de-volatilized by heating to 150°C under 250 mmHg (33.3 kPa) and a nitrogen flow (0.5 standard cubic feet per minute, scfm (0.47standard l/s)). The speed of the agitator was set at 100 rpm. Tin ethylhexanoate (54.93 g) was added, and the reaction mixture was heated to 195°C under atmospheric pressure and a nitrogen flow of 1.2 scfm (0.57standard l/s). The pressure was reduced to 500 mmHg (66.7 kPa) and the reaction was continued for another 2 hours. A polyol having a hydroxyl number of 87 was obtained, based on the sunflower-monomer alcohol. The procedure of ASTM 4274 was used to determine hydroxyl number. Properties of the polyol are shown in Table 2.

Table 2: Polyol Preparation and Properties[1]

Ex. #	FAME (kg)	Initiator INT-1 (kg)	M / I mole ratio	Catalyst (g)	Temp (°C)	Run Time (hr)	Viscosity (cP at 25°C)	Hydroxyl number
1	Sun 37.875	17.050	4.1	Sn(II) (54.93)	195	2	1620	87

1. FAME" refers to fatty acid methyl esters. "M/I ratio" indicates the molar ratio of hydroxymethylated fatty acid methyl ester monomer (M) to initiator (I). "Sn(II)" refers to tin ethylhexanoate.

General Procedure for the Production of Polyurethanes from Sunflower Oil-Based Polyols

A polyurethane foam was prepared according to the following general procedure. Chemical components used for the preparation of the polyurethane foam included the following materials.

Voranol 3512, which is a 2.55 average functional, 12.3 wt. percent ethylene oxide, heterofed 3,500 MW polyol, was obtained from The Dow Chemical Company.

Water used for these formulations was distilled, deionized water.

NIAX L-703 is a silicone surfactant obtained from General Electric.

NIAX A-1 is an amine urethane blowing catalyst obtained from General Electric.

DABCO T-9, a stabilized stannous octanoate, is a catalyst used in flexible slabstock foams and is obtained from Air Products and Chemicals, Inc.

Voranate T-80 is type I TDI (toluene diisocyanate) with an equivalent weight of 87. Used in making flexible foams, it was obtained from The Dow Chemical Company.

All of the polyol components, the water, surfactant, and blowing catalyst of the given formulation were individually metered and weighed into a one quart capacity metal cup. The contents were premixed for 15 seconds at 1800 rpm using a pin type mixer. The tin catalyst, dispensed by volume, was then added to the stirred components and mixed for an additional 15 seconds at 1800 rpm. A stoichiometric amount of toluene diisocyanate (Voranate T-80) was then added to the cup and vigorously mixed for 3 seconds at 2400 rpm. The cup contents were then poured into a 15 inch x 15 inch x 10 inch (38.1 cm x 38.1 cm x 25.4 cm) wooden box lined with a polyethylene bag. The blowoff time and any other distinct reaction characteristics were recorded. The cream time was 14 seconds, and the rise time was 124-125 seconds. The foam buns were allowed to cure overnight under a ventilated fume hood. The buns were then placed in ambient storage and submitted for physical property assessment using ASTM test method designation D 3574-95.

According to the general procedure, the foams were prepared using the following formulations with the results of mechanical testing included in Table 3.

Table 3 – Preparation and Physical Properties of Polyurethane Foams

Example #	2	3	4
Monomer Alcohol Diol/Triol Wt. Ratio	**5.56**	**5.56**	**5.56**
V-3512	50	50	50
Sunflower Polyol	50	50	50
Water	3.0	3.0	3.0
NIAX L-703	1.00	1.00	1.00
NIAX A-1	0.10	0.10	0.10
DABCO T-9	0.12	0.14	0.16
Voranate T-80 TDI	41.50	41.50	41.50
Index[A]	105	105	105
Properties			
Air Flow, CFM	3.8	2.6	1.6
Compression Set, 90%	5.9	4.7	6.5
Density (kg/m^3)	28.5	28.5	26.0
IFD, 40%	128	136	140
Resiliency, %	37	37	35
Tensile (kPa)	68	67	82
Tear (N/cm)	512	520	507
Elongation, %	191	185	191

A. "Index" refers to the stoichiometric excess of isocyanate to all hydroxy-containing components of the formulation, i.e., [NCO]/[OH] x 100. An index of 105 means that 5 molar excess of isocyanate is present over all OH equivalents.

From Table 3 it is seen that polyols prepared from a monomer alcohol having a diol/triol weight ratio of 5.56/1. within the scope of the claims and close to the lower claimed limit of 5/1, produced foams having measurable properties within a range acceptable for use in flexible foam applications.

Remarks Concerning Intl. Patent Application PCT/US07/86222

In Affiant's Declaration I Under Rule 132, filed originally on November 26, 2007, and slightly revised and re-filed concurrently herewith, the Affiant provided comparative experiments CE-1 to CE-5 showing that when an alcohol composition having a diol/triol weight ratio less than 5/1 was used, downstream polyurethane polymers were formed with tin splits and thus were unsuitable for flexible foam applications. After filing the instant application, Affiant's research team at The Dow Chemical Company (the same employer of the present Applicants) discovered that suitable flexible polyurethane foams could be prepared from alcohol compositions having a diol/triol weight ratio less than 5/1, provided that a new parameter was taken into account. The new parameter was defined as an "average functionality number (AFN);" and to obtain good flexible foams, the AFN had to be fixed within a narrow range of greater than 0.96 to less than 1.26. This later discovery became the subject of International Patent Application PCT/US2007/86222, cited concurrently herewith, of which Affiant is also a co-inventor.

With respect to the instant application, the Affiant believes that the comparative experiments CE-1 to CE-5 provided in the first Declaration, using an alcohol having a diol/triol weight ratio less than 5/1 that failed at making flexible foams, were a fair and honest representation of the prior art at the time the present invention was made, and that any advancement in preparing successful flexible foams using an alcohol having a diol/triol weight ratio less than 5/1 constitutes a second invention originating in Affiant's own research team.

The undersigned DECLARANT declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application of any patent issuing thereon.

April 28, 2008 — Date

David A. Babb